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ION IMPLANTATION OF WIDE BANDGAP SEMICONDUCTORS

C.L. Anderson, H.L. Dunlap, C.L. Ramiller, and G.S. Kamath

Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

November 1977

Contract No. N00173–77–C–0051
Annual Technical Report No. 1
For Period 1 October 1976 through 30 September 1977

Sponsored By
NAVAL ELECTRONIC SYSTEMS COMMAND
Department of the Navy
Washington, D.C. 20360

Monitored By
NAVAL RESEARCH LABORATORY
Department of the Navy
Washington, D.C. 20375

HUGHES

HUGHES AIRCRAFT COMPANY
RESEARCH LABORATORIES

(6) ION IMPLANTATION OF WIDE BANDGAP SEMICONDUCTORS

(9) Annual Technical Report No. 1
for Period Covering
1 Oct [redacted] — 30 Sep [redacted] 77

(15) CONTRACT N00173-77-C-0051

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(11) NOV[redacted]77

(12) 40 p.

(10) Prepared by

C.L. Anderson, H.L. Dunlap, C.L. Ramiller [redacted] G.S. Kamath

172 600

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) ION IMPLANTATION OF WIDE BANDGAP SEMICONDUCTORS		5. TYPE OF REPORT & PERIOD COVERED Annual Technical Report 1 1 Oct 1977-30 Sep 1977 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) C.L. Anderson, H.L. Dunlap, C.L. Ramiller, and G.S. Kamath		8. CONTRACT OR GRANT NUMBER(s) N00173-77-C-0051
9. PERFORMING ORGANIZATION NAME AND ADDRESS Hughes Research Laboratories 3011 Malibu Canyon Road Malibu, CA 90265		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Department of the Navy Arlington, VA 22217		12. REPORT DATE November 1977 13. NUMBER OF PAGES 39
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) Naval Research Laboratory Department of the Navy Washington, D.C. 20375		15. SECURITY CLASS. (of this report) 15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Reproduction in whole or in part is permitted for any purpose of the United States government.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

GaAs ion implantation, GaAs encapsulation, GaAs annealing.

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The principal dopants studied under this program are sulfur and selenium, both n-type. Topics covered in this report include (1) encapsulation technology, (2) transferability of sulfur or selenium implantation technology, (3) dual implantation of sulfur or selenium with gallium, and (4) thermal conversion of semi-insulating GaAs.

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ACKNOWLEDGEMENT

We wish to thank the Electrical Engineering Department and Kellogg Radiation Laboratory of the California Institute of Technology for making their backscattering facilities available for use in these experiments.

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SECTION 1

INTRODUCTION AND SUMMARY

The primary goals of this program are to develop an understanding of the basic physical processes that control the electrical characteristics of ion-implanted layers in GaAs and to exploit the understanding gained to develop a transferable technology for reliable ion-implantation doping of GaAs. During the one-year contract period covered by this report, a major effort was made to study, through duplicate processing and evaluation projects at Hughes Research Laboratories (HRL) and the Naval Research Laboratory (NRL), the factors that affect the transferability and reproducibility of implanting sulfur and selenium into GaAs using Si_3N_4 as an annealing encapsulant.

At the outset of this period, neither HRL nor NRL had the capability to deposit the Si_3N_4 films required for the transfer tests. Accordingly, both laboratories attempted to develop the required deposition capability. Using Hughes internal funding, we constructed a general-purpose atmospheric-pressure chemical vapor deposition (CVD) system capable of depositing Si_3N_4 and a variety of other materials. Adherent, stoichiometric Si_3N_4 films have been prepared in this system. Despite their excellent properties as barriers to Ga diffusion, these films have not been successfully used to encapsulate ion-implanted GaAs. We investigated their use in conjunction with pyrolytic SiO_2; we also studied the properties of silicon oxynitrides in several compositions as encapsulants for GaAs. The most successful material at HRL was SiO_2 prepared by pyrolysis of silane at 390 to 420°C. SiO_2 prepared by pyrolysis of silane in nitrous oxide at 600°C is considerably superior as a Ga diffusion barrier to the same material deposited at 420°C, and shows some promise as an encapsulant. The results of our studies of encapsulant properties are presented in Section 2.

Before Si_3N_4 films were available at either HRL or NRL, we completed a test of the transferability of sulfur implantation into GaAs, using Si_3N_4 films supplied by the LFE Corporation, Waltham, MA. This project, performed jointly by HRL and NRL, demonstrated that statistically

equivalent electrical data can be obtained from samples prepared independently at our two laboratories when the same encapsulant is used on all the test samples. The results of this test are summarized in Section 3.

The second area investigated was the use of dual implants (e.g., S+Ga) to correct for implant-induced stoichiometry imbalances, particularly at high implant fluences. We attained a 40% (relative) increase in the electrical activity of selenium implants at the 10^{14} cm^{-2} fluence level. No similar increase occurred for sulfur implants. The results of these experiments are summarized in Section 4.

The reliable use of semi-insulating GaAs substrates for ion implantation and device purposes requires that the substrates exhibit high thermal stability. The resistivity of most semi-insulating GaAs material is significantly reduced after high-temperature annealing. NRL has been exploring the possibility of growing high-purity semi-insulating GaAs by the liquid-encapsulated Czochralski (LEC) process. Hughes has developed a simple, reproducible technique for evaluating the thermal stability of GaAs wafers. During this report period we evaluated several samples of the NRL LEC material and samples of material from other sources supplied by NRL. The results of these studies are presented in Section 5.

SECTION 2

SILICON NITRIDE AND SILICON OXYNITRIDE FILMS FOR ENCAPSULATION OF GaAs

The use of pyrolytic silicon nitride and silicon oxynitride films as encapsulants for GaAs was examined in some detail. This work was funded both by the present contract and Hughes internal funding. All of this work is summarized here. Briefly, contract funding was used only for deposition evaluation of Si_3N_4 films. Internal funding was used in constructing the deposition system and in all work with silicon oxynitrides.

The work reported here involves films deposited in a cold-walled, atmospheric-pressure CVD system. The system is highly automated and helium leak-tight to maximize reproducibility and reduce contamination. The silicon nitride process used in this reactor is similar to that used by MIT Lincoln Laboratories[1] in the sense that the films are deposited by pyrolysis of silane and ammonia and the sample temperature is raised rapidly to the 600 to 700°C range for deposition. In contrast to the Lincoln Labs system (which employs a strip heater), our system uses an rf-heated susceptor on which we have simultaneously encapsulated as many as 40 typical GaAs samples (roughly 7 mm x 7 mm). The temperature of the susceptor can be raised from 400°C to 700°C in less than 10 sec. Using this system, we have successfully deposited Si_3N_4 using nitrogen and argon as carrier gases and SiO_2 and SiO_xN_y using a nitrogen carrier. The resulting films have been analyzed using Rutherford backscattering (RBS) and Auger electron spectroscopy to determine thickness, stoichiometry, and the presence of contaminants. The Si_3N_4 films grown using a nitrogen carrier were stoichiometric and free of oxygen and other impurities. Si_3N_4 films grown using an argon carrier, however, contained approximately 9% oxygen (apparently because oxygen was present in the in-house argon supply) but were free of other impurities. All work reported below involved films prepared with nitrogen carrier gas.

The first test of our Si_3N_4 films for encapsulating ion implants into GaAs used films roughly 3000 Å thick. These films were applied to wafers implanted with selenium (overall implant 5×10^{12} cm^{-2} 260 keV Se^+, at room temperature or 250°; corner contacts: 5×10^{14} cm^{-2} 20 keV S^+, 5×10^{14} cm^{-2} 140 keV S^+ at room temperature). These samples were intended to be used in a test of the transferability of the selenium ion implantation process in GaAs (transfer test "Se 1").[2] After annealing of these samples at 800°C, Hall-effect measurements indicated that the electrical properties of the implanted layers were extremely variable (see Table 1). Because of these problems, the transferability of this process was not evaluated. Visual examination of the wafer surfaces revealed easily visible striped patterns. These were initially believed to result either from incomplete removal of the encapsulant or from localized etching of the wafer surface during encapsulant removal. Accordingly, one sample (128) was treated further by removing the Ohmic contacts with HCl, soaking in HF to remove any residual Si_3N_4, and reapplying the Ohmic contacts. Neither the optical nor electrical properties of the sample were changed significantly. Four additional samples (123, 124, 126, 127) were treated further simply by removing and reapplying the contacts. Two of these samples exhibited minor changes in properties after this treatment. The sheet resistance of one sample (127) nearly doubled with no change in sheet carrier concentration. This is not particularly remarkable since the electrical asymmetry of the sample was high (resistance ratio ≃ 300-400). Sample 126 dramatically increased in asymmetry after this process (resistance ratio change from 240 to 4×10^4). Apparently, the treatment of this sample destroyed one of the corner contacts.

These results indicated that the appearance of the sample was not the result of residual encapsulant on the surface. Although we had not seen any evidence of stress-related effects in our experiments with unimplanted wafers, we attributed the stripe patterns observed on the samples to damage caused by stress in the nitride coating. As shown in Figures 1 and 2, the damage typically consists of perpendicular cracks or ridges over the face of the <100> slice. Stoichiometric

Table 1. HRL Results

Selenium Transfer Test "Se 1", 800° Anneal

Implant Temperature, °C	Die Number	Sheet Resistivity, (Ω/□)	Hall Electron Mobility $cm^2\ V^{-1}\ sec^{-1}$	Sheet Electron Concentration, cm^{-2}	Resistance Ratio
20 (Nominal)	123	469	3960	3.4×10^{12}	4.3
	123[a]	453	4250	3.2×10^{12}	1.6
	124[c]	2.3×10^{7}	-14.2	-1.9×10^{10}	4×10^{6}
	124[a,c]	2.6×10^{7}	-0.29	-8.3×10^{11}	2×10^{5}
	126[c]	3.2×10^{7}	-0.89	-2.2×10^{11}	240
	126[a,c]	1.5×10^{7}	-10.6	-3.9×10^{10}	4×10^{4}
	127	654	3140	3.0×10^{12}	370
	127[a]	1210	1720	3.0×10^{12}	270
	128	1000	2400	2.6×10^{12}	160
	128[b]	1070	2630	2.2×10^{12}	78
	129[a]	2.3×10^{8}	22.0	1.2×10^{9}	2.4
	130[a]	4.7×10^{7}	12.3	1.1×10^{10}	19
250	133[a]	8590	832	8.7×10^{11}	15
	134[a]	330	1480	1.3×10^{13}	9.2
	135[a]	5300	173	6.8×10^{12}	3×10^{4}
	136[d]	370	3240	5.2×10^{12}	1.4
	137[a]	1.6×10^{8}	10.4	3.7×10^{9}	9.6
	138	263	2440	9.7×10^{12}	190
	139[a]	9.0×10^{6}	53.8	1.3×10^{10}	1×10^{6}
	140	216	3960	7.3×10^{12}	2.5
20 (Control)	131	1.7×10^{5}	-54.0	-6.8×10^{11}	30
	132	1.2×10^{5}	-82.1	-6.2×10^{11}	1.2
250 (Control)	141[a]	1.6×10^{7}	2.91	1.4×10^{11}	360
	142[a]	2.0×10^{7}	19.5	1.6×10^{10}	1.1

[a] Remeasured after contact removal with HCl and then reapplied.

[b] Remeasured after contacts removed with HCl, HF soak, and contact reapplication.

[c] Measurement problems encountered (negative resistance or large drifts).

[d] Sample prepared with different coating sequence.

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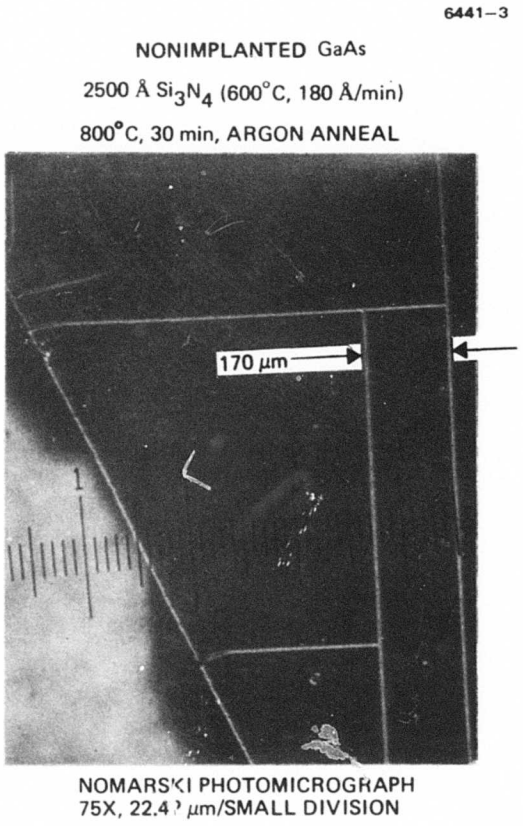

Figure 1. Nomarski photomicrograph near the edge of a nonimplanted GaAs sample encapsulated with 2500 Å of Si_3N_4 at 600°C and annealed at 800°C for 30 min.

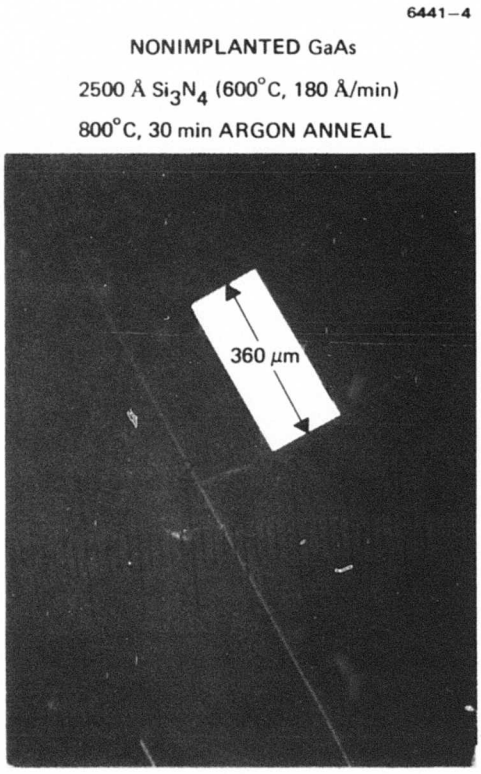

Figure 2. Nomarski photomicrograph at the center of a nonimplanted GaAs Sample encapsulated with 2500 Å of Si_3N_4 at 600°C and annealed at 800°C for 30 min.

Si_3N_4 films deposited by pyrolysis of silane and ammonia are under high tensile stress, which is intrinsic and not due to thermal mismatch.[3] Since this stress varies with deposition parameters, we carefully characterized Si_3N_4 deposition in our CVD system.[4] We began with our 600°C Si_3N_4 process since at this temperature there is usually no obvious deterioration of the back of the dice from crystal decomposition during nitride deposition. Figure 3 gives film thickness as a function of deposition time at 600°C for the same total gas flow, silane concentration, and ammonia concentration used last quarter to deposit Si_3N_4 at 700°C. The scatter presumably results because (1) we do not have mass flow controllers for the system, and (2) nonuniformities in gas flow and susceptor temperature produce noticeable variation in film thickness across most wafers. Figure 4 shows growth rate as a function of silane concentration at 600°C with constant total flow and ammonia concentration. In all cases, the NH_3:SiH_4 ratio was well above the 10:1 ratio required to produce stoichiometric Si_3N_4 at these temperatures.[3] At a given deposition temperature, the intrinsic stress in the film can be lowered by reducing the growth rate.[3]

The second test of our pyrolytic Si_3N_4 films for implant encapsulation used samples implanted with 1×10^{13} cm^{-2} Se^+ implanted at 260 keV and ambient temperature.[4] All samples received contact pads of 5×10^{13} cm^{-2} Se^+ at 220 keV. Several dice were reserved as control samples after this step and were not given the overall implant. The dice were processed in batches of six for each encapsulation procedure (four test dice and two control samples per batch). Each batch was treated with a different encapsulant process. The silicon nitride used in this study was deposited at growth rates between 30 Å/min and 180 Å/min. The films used for transfer test "Se 1" were deposited at 700°C at a rate of 1000 Å/min. Consequently, the stress levels in these films should be considerably lower than those used for test "Se 1". All dice were processed identically with regard to cleaning, annealing, and measurement procedures. Annealing was performed in titanium-gettered argon for 30 min at 800°C.

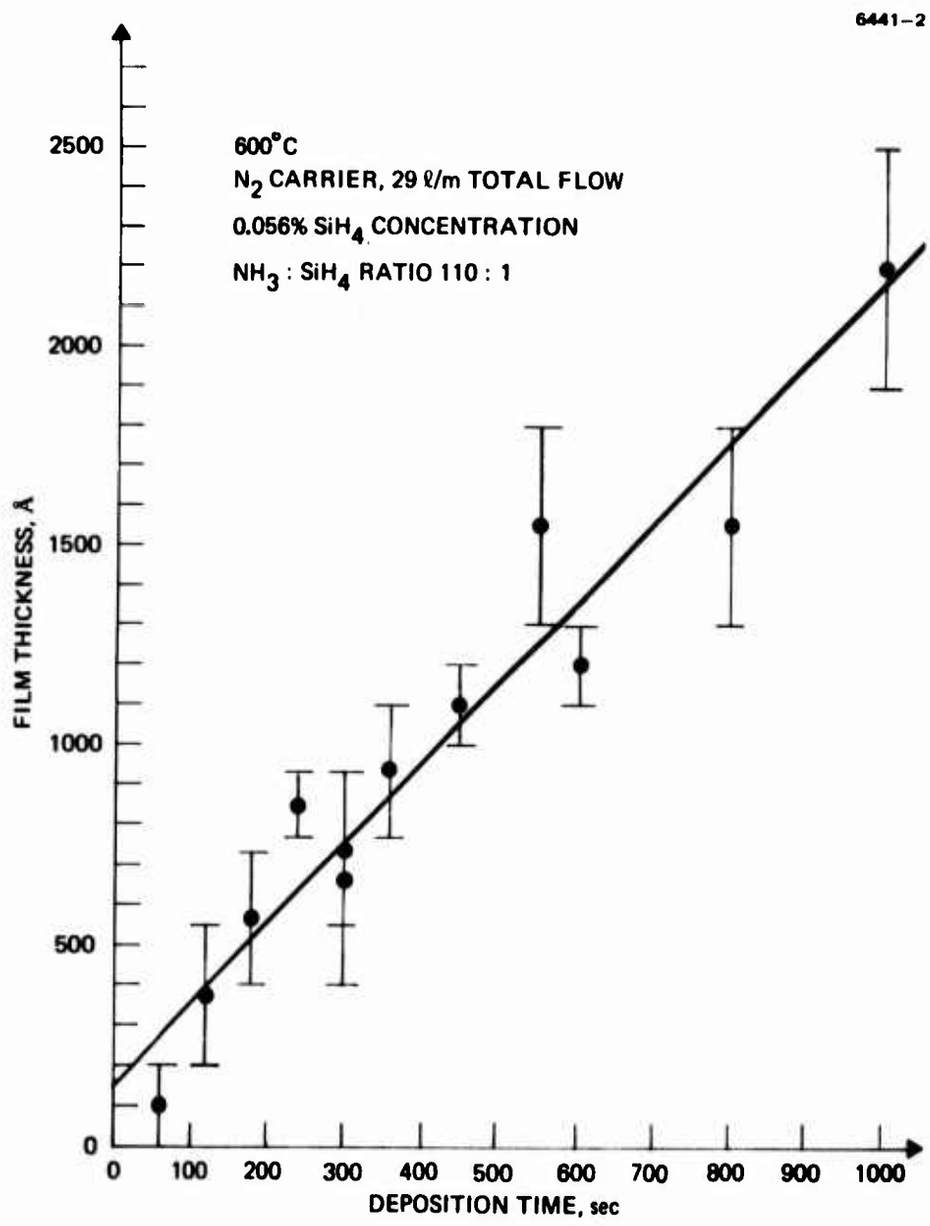

Figure 3. Film thickness as a function of deposition time for 600°C pyrolytic Si_3N_4; N_2 carrier, 29 liters/min total flow, 0.056% SiH_4 concentration, NH_3:SiH_4 ratio 110:1.

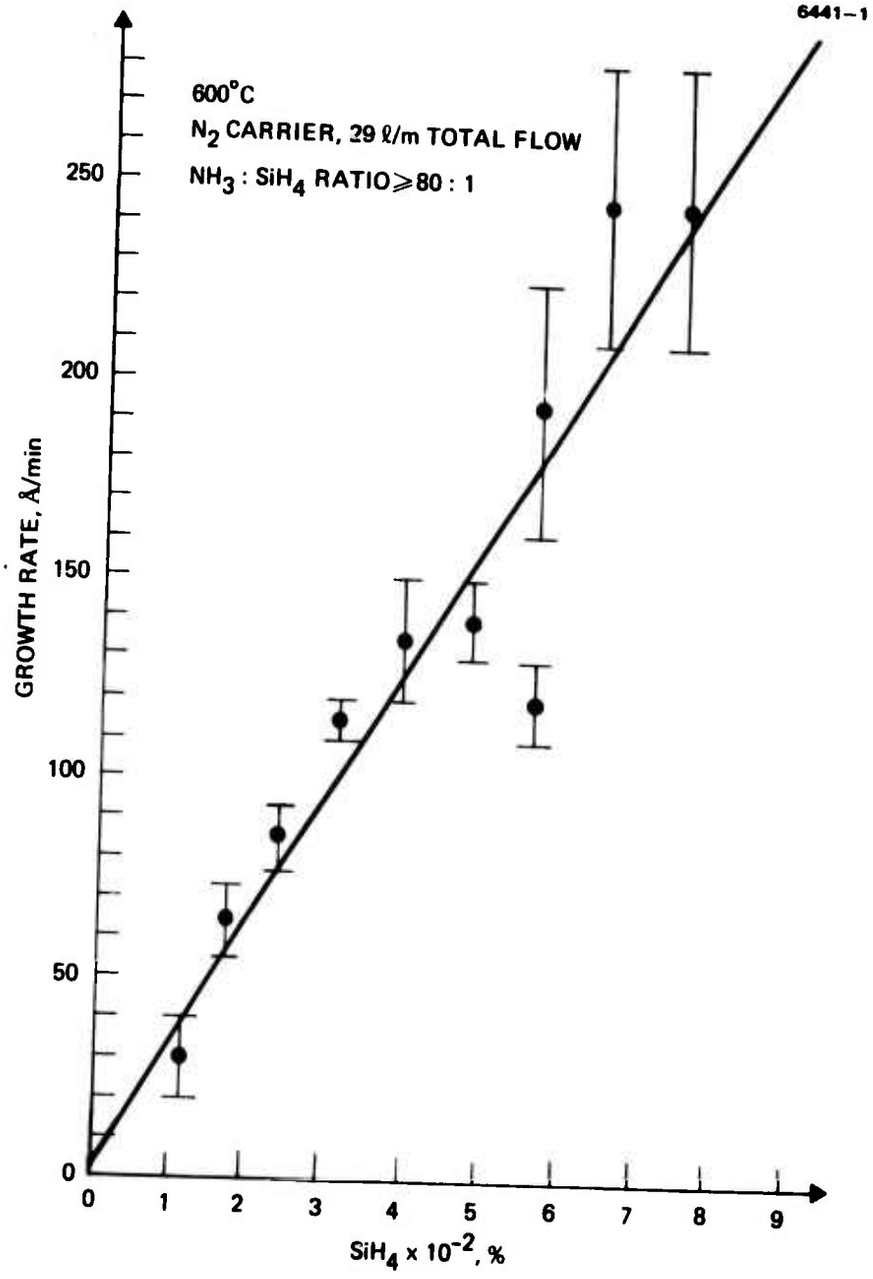

Figure 4. Growth rate as a function of silane concentration for 600°C pyrolytic Si_3N_4; N_2 carrier, 29 liters/min total flow, NH_3:SiH_4 ratio $\geq$ 80:1.

Table 2 summarizes the results of experiments done so far to determine whether surface damage could be eliminated by changing deposition parameters. Unimplanted GaAs and Se-implanted GaAs were coated with various encapsulants and then annealed at 800°C in argon for 30 min. None of the SiO_2 films caused any visible damage. This is consistent with the hypothesis that the problem is stress related. SiO_2 films deposited on Si are typically under a tensile stress of 1 to 4 x 10^9 dyn/cm^2 (Ref. 5,6), although Si_3N_4 films on Si are under a tensile stress of 0.5 to 1.5 x 10^{10} dyn/cm^2 (Ref. 3). The thicker (2200 to 2500 Å) Si_3N_4 films caused visible damage to GaAs samples whether or not implanted, even when an oxide-coated interface was used. The structures that seem to have the most promise are (1) very thin (∿200 Å) Si_3N_4 films over the implant followed by SiO_2 on both sides of the samples, (2) oxynitrides, and (3) very thin SiO_2 followed by thin Si_3N_4 over the implant and then by SiO_2 on both sides. We believe that the thick Si_3N_4 layer on top of the thin oxide was responsible for the damage observed in batch E.

Table 2. Characterization of Visible Surface Damage to GaAs and Se-implanted GaAs Substrates Annealed at 800°C in Argon for 30 min

Batch	Encapsulation	Not Implanted	Se-Implanted
A	SiO_2, HRL-EB, 3000 Å	No damage	No damage
B	SiO_2, HRL-II, 2700 Å, 400°C	No damage	No damage
C	Si_3N_4, 2500 Å, 600°C, 180 Å/min on the front, then SiO_2, 2500 Å, 390°C both sides	Damaged	Damaged
D	Si_3N_4, 200 Å, 600°C, 30 Å/min on the front, then SiO_2, 2500 Å, 390°C both sides	No damage	No damage
E	SiO_2, HRL-II, 200 Å, 390°C, under Si_3N_4, 2200 Å, 600°C, 130 Å/min on the front, then SiO_2, 2500 Å, 390°C both sides	Damaged	Damaged
F	Si_3N_4, 500 Å, 600°C, 180 Å/min on the front, then SiO_2, 2500 Å, 410°C both sides	No damage	No damage

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Table 3 presents the results of gallium in-diffusion experiments performed under internal funding. Films of Si_3N_4, SiO_2, SiO_xN_y, Si_3N_4 over SiO_2, and SiO_2 over Si_3N_4 were deposited in our pyrolytic CVD system (denoted HRL-II). The Si_3N_4 films were stoichiometric and did not contain any oxygen detectable by RBS or AES. Additional samples of SiO_2 on Si were prepared at Hughes Torrance Research Center (TRC) and in another CVD system at HRL (denoted HRL-EB). One of the TRC samples was "densified" by annealing in O_2 at 1000°C. All of the samples were submerged under gallium in a carbon boat and annealed at 800°C in a clean argon atmosphere. After cooling, the samples were washed in HCl to remove any gallium on the surface. Backscattering analysis was then performed to determine the concentration and distribution of gallium in the films.

Table 3. Gallium In-diffusion Results

Films Deposited	Result of RBS Measurements
SiO_2, HRL-EB, 3300 Å	Ga diffused throughout the film. Highest concentration was in the top one third.
SiO_2, TRC, 1200 Å	Ga diffused throughout the film. Concentration was fairly uniform.
SiO_2, TRC, 1500 Å "densified"	No Ga diffusion.
SiO_2, HRL-II, 3000 Å, 380°C	Ga diffused throughout the film. Highest concentration was in the top one third.
SiO_2, 1100 Å, 390°C under Si_3N_4, 550 Å, 640°C	No Ga diffusion.
Si_3N_4, 650 Å, 640°C under SiO_2, 1100 Å, 390°C	Ga diffused into the SiO_2 as far as the SiO_2/Si_3N_4 interface.
Si_3N_4, 1250 Å, 700°C	No Ga diffusion.
$SiO_{1.8}N_{0.5}$, 2000 Å, 680°C	Ga diffused throughout the film. Concentration was the highest of this group of samples.

Note: Si substrates, annealed under gallium at 800°C for 30 min.

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Only two effective diffusion barriers were identified during this study: Si_3N_4 and "densified" SiO_2. Although the latter barrier would not be practical for use on GaAs unless the densification temperature could be lowered, it does demonstrate that a dense SiO_2 film can block gallium diffusion. The poorest diffusion barrier in the group was an oxygen-rich oxynitride of approximate composition $SiO_{1.8}N_{0.5}$. Because GaAs diffuses through a layer of SiO_2 and then stops at an Si_3N_4 layer, we also decided to try a thin SiO_2 film as an interface between the GaAs and the Si_3N_4 in the hope that it would reduce the stress on the surface and perhaps saturate with a small amount of gallium.

Table 4 summarizes the electrical properties of the Se-implanted samples as a function of encapsulant treatment, indicating the average and standard deviation of the electrical measurements. All of the dice are from the same ingot: Cr-doped semi-insulating GaAs No. 2988 from Crystal Specialties. These results show that interface contact with even the thinnest nitride layer (200 Å) is sufficient to strain the surface and decrease carrier mobility. As suggested by the SiO_2-encapsulated control dice, there may be a residual doping level in this material that would interfere with experiments at low fluence levels but not with encapsulant comparisons.

Since the results with stoichiometric Si_3N_4 films were disappointing, our most recent investigations have concentrated on determining the effects of oxygen contamination on the encapsulant properties of our films. Internally funded investigation of the dielectrics (for possible use on GaAs) has given us the capability to deposit SiO_xN_y films at 600°C with control of the oxygen content. This allows studying the effect of oxygen in the films on Ga in-diffusion from a melt, Ga out-diffusion from GaAs substrates, and the electrical properties of Se-implanted GaAs.

The deposition process uses a mixture of silane, ammonia, and nitrous oxide; this process and the properties of the films produced have been studied previously.[7,8] For the samples used in this study, a total N_2 carrier gas flow of 30 liters/min was used with the silane and ammonia flows held constant at 11 cm^3/min and 6 liter/min, respectively;

Table 4. Electrical Measurements

Batch	Encapsulation	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/V\text{-}sec$	Sheet Electron Concentration, cm^{-2}
A — implanted	SiO_2, HRL-EB, 3000 Å	454 ± 32	3360 ± 270	$(4.10 \pm 0.18) \times 10^{12}$
— control		1520 ± 180	4036 ± 93	$(1.03 \pm 0.14) \times 10^{12}$
B — implanted	SiO_2, HRL-II, 2700 Å, 400°C	402 ± 40	3210 ± 350	$(4.88 \pm 0.17) \times 10^{12}$
— control		980 ± 160	4130 ± 210	$(1.57 \pm 0.34) \times 10^{12}$
C — implanted[a]	Si_3N_4, 2500 Å, 600°C, 180 Å/min on the front, then SiO_2, 2500 Å, 390°C on both sides	$(4 \pm 4) \times 10^8$	91 ± 77	$(1.3 \pm 1.7) \times 10^9$
— control[a]		$(1.4 \pm 0.6) \times 10^8$	238 ± 68	$(2.4 \pm 1.6) \times 10^7$
D — implanted	Si_3N_4, 200 Å, 600°C, 30 Å/min on the front, then SiO_2, 2500 Å, 390°C on both sides	1020 ± 260	2370 ± 670	$(2.70 \pm 0.10) \times 10^{12}$
— control		$(5.7 \pm 1.2) \times 10^8$	682 ± 76	$(1.65 \pm 0.18) \times 10^7$
E — implanted[a]	SiO_2, HRL-II, 200 Å, 390°C, under Si_3N_4, 2200 Å, 600°C, 130 Å/min on the front, then SiO_2, 2500 Å, 390°C on both sides	$(2.91 \pm 1.5) \times 10^8$	160 ± 80	$(2.9 \pm 3.7) \times 10^8$
— control[a]		$(1.7 \pm 0.5) \times 10^8$	164 ± 53	$(2.6 \pm 1.5) \times 10^8$
F — implanted	Si_3N_4, 500 Å, 600°C, 180 Å/min on the front, then SiO_2, 2500 Å, 410°C on both sides	$(1.4 \pm 0.9) \times 10^4$	140 ± 190	$(4.6 \pm 3) \times 10^{12}$
— control		6.00×10^7[b]	14.45	7.12×10^9

Note: [a]Visible disruption of implanted surface.

[b]Only one sample available.

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the N_2O flow was increased from 0 to 550 cm^3/min. In Figure 5, ternary diagram indicates with a solid line the expected compositions ranging between Si_3N_4 and SiO_2 if SiO_xN_y is a pseudo-binary mixture of Si_3N_4 and SiO_2. The data shows the composition of the first series of films produced. The slight deficiency of Si in our films compared to the pseudo-binary is consistent with the results of others.[9] The composition was determined by depositing the films on Si substrates that had previously been coated with pyrolytic carbon and then performing 2 MeV RBS measurements. The carbon films were thick enough to shift the portion of the spectrum due to the Si substrate to energies well below those of the N and O peaks so that the composition could be determined more accurately.

The same series of SiO_xN_y films was simultaneously deposited on clean <100> Si substrates, and the substrates were subsequently annealed under liquid Ga at 800°C for 30 min. The samples coated with Si_3N_4 or with SiO_xN_y containing 21 or 28 at.% O did not survive the anneal. Portions of RBS spectra of the remaining samples showing the relative amounts of Ga that diffused into the different films are shown in Figure 6. As the oxygen content of the films is increased, they become even less effective as a diffusion barrier to Ga than is SiO_2. The permeability of the films to Ga apparently increases with the oxygen concentration, but somewhere in the range of 46 to 66 at.% the solubility of Ga in the films begins to decrease. The SiO_2 films used here were produced from silane and nitrous oxide at 600°C rather than from silane and oxygen at 390°C, as in our usual silox process.

In Figure 7, RBS spectra are shown for a GaAs substrate capped with SiO_xN_y containing 47 at.% O and for a GaAs substrate capped with 600°C SiO_2. Both were annealed in forming gas at 800°C for 30 min. The GaAs substrate edge is at a much lower energy in the case of the SiO_2 film because that film was considerably thicker than the oxynitride. Much more out-diffusion occurs into the oxynitride than into the SiO_2, which is consistent with the results of the in-diffusion experiments. The assumption that the diffusing species is Ga based on previous work.[10] This will be confirmed using Auger spectroscopy.

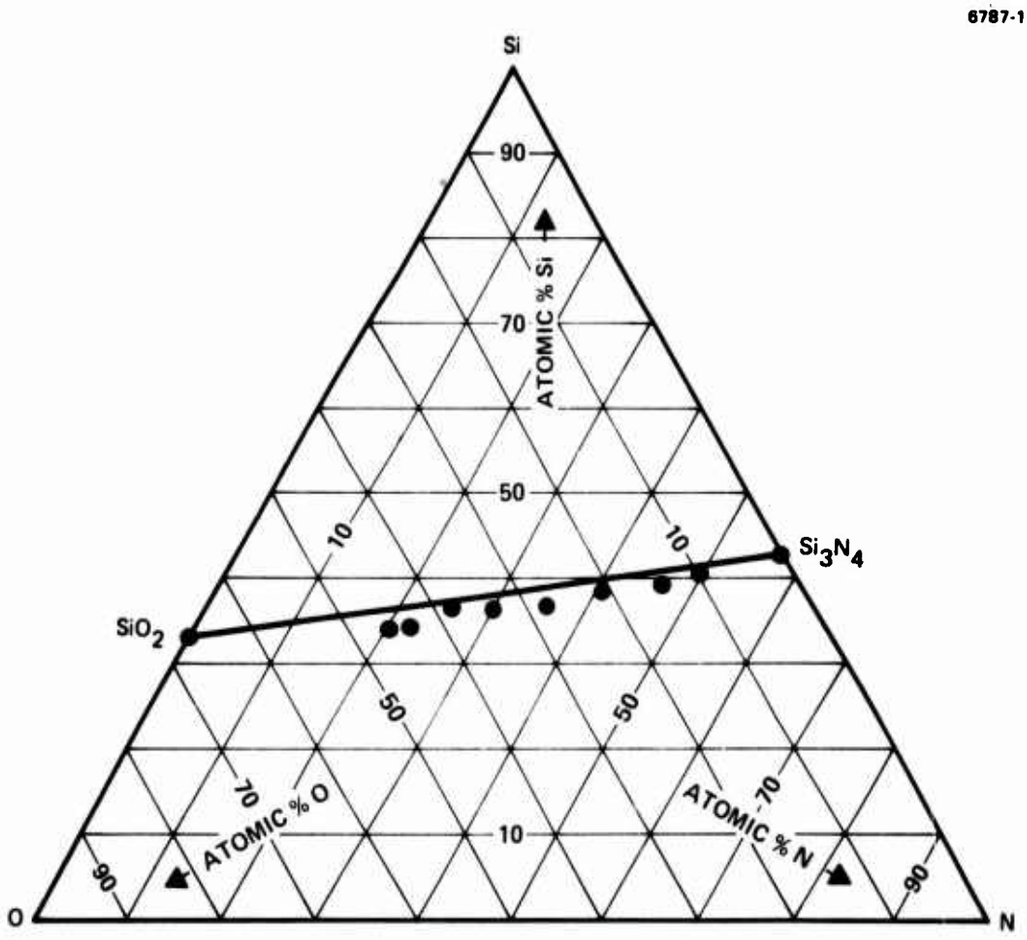

Figure 5. Composition of pyrolytic silicon oxynitrides deposited at 600°C using silane, ammonia, and nitrous oxide (nitrogen carrier).

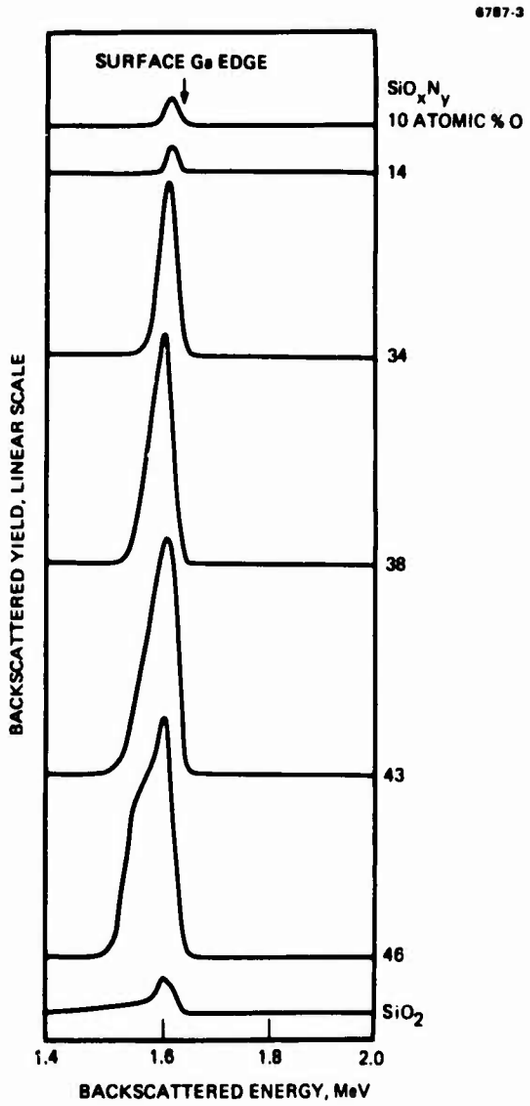

Figure 6. Gallium diffusion into SiO_xN_y and SiO_2 films deposited at 600°C. The curves show only the portion of the 2NeV $^4He^+$ backscattering spectra caused by Ga in the deposited films.

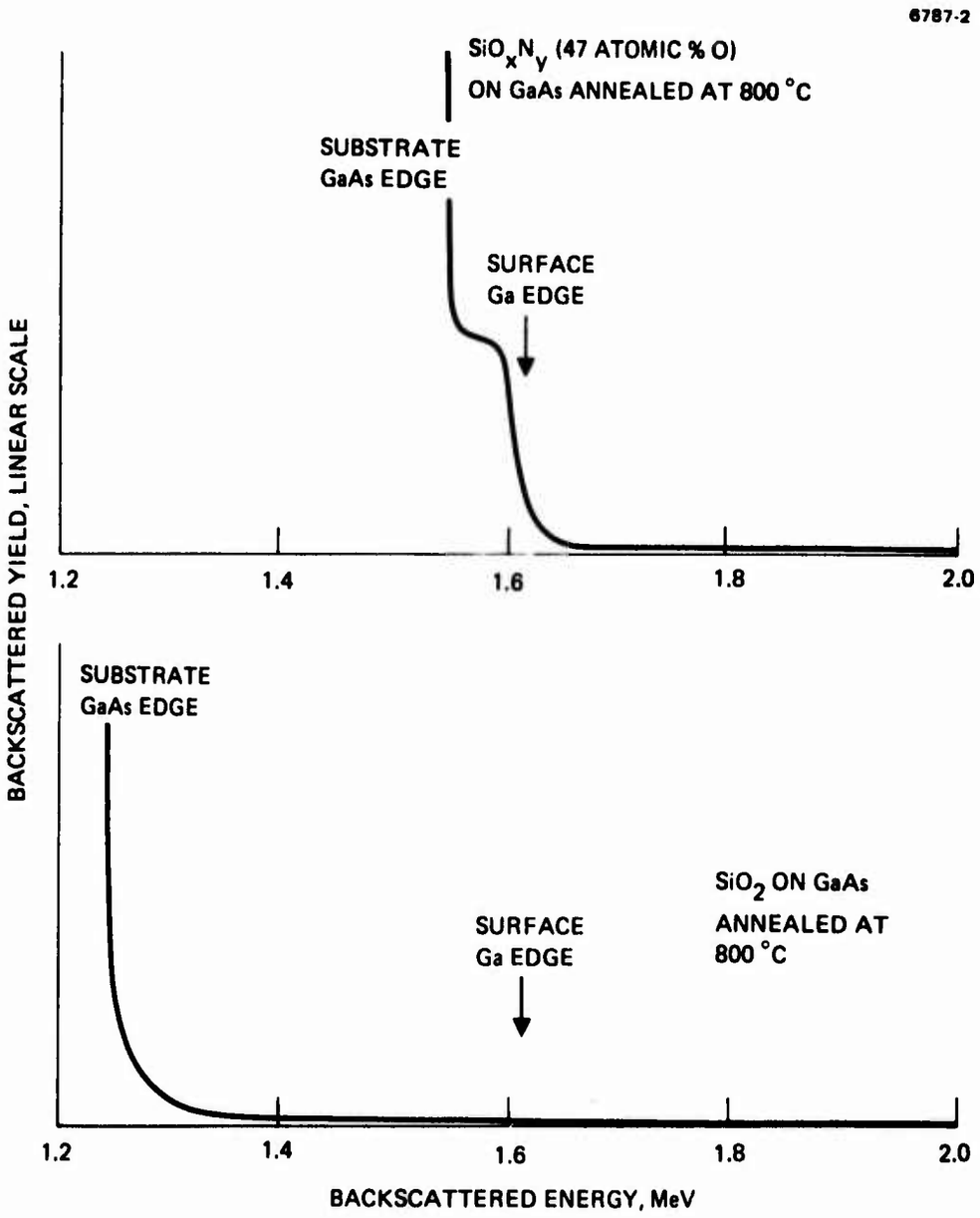

Figure 7. Comparison of Ga out-diffusion from GaAs as determined by 2MeV $^4He^+$ backscattering measurements.

Another series of films ranging in composition from Si_3N_4 to SiO_2 has been deposited on samples of Si, carbon-coated Si, and GaAs. RBS determination of the composition of each film is complete, and the Ga out-diffusion from a set of GaAs substrates annealed at 800°C for 30 min is being measured. Table 5 gives the physical appearance of a set of GaAs samples that were annealed at 900°C for 30 min in cleaned argon. Examining the samples under a dark field microscope showed that films of Si_3N_4 and oxynitride containing up to 9 at. % O exhibited cracking, while films containing 21 to 47 at. % O were either blistered or contained many bubbles or defects visible at low magnification. Only the 600°C SiO_2 film appeared free of significant damage; it contained only a few very small bubbles or precipitates.

Table 5. Physical Appearance of Encapsulated GaAs Samples after a 900°C, 30-min Anneal

Atomic % O	Appearance
0 (Si_3N_4)	cracked
7	cracked
9	cracked
21	intact but contains many defects
30	intact but contains many defects
36	intact but contains many defects
43	blistered
45	blistered
45	blistered
47	blistered
66 (SiO_2)	small number of bubbles, or precipitates.

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Our most recent evaluations of encapsulant effects on the electrical properties of ion-implanted layers have employed selenium implants into two semi-insulating (Cr-doped) ingots. All dice described here were provided with implanted corner contact pads (5×10^{13} 200 keV Se^{+}/cm^{2} implanted at room temperature). Some dice were reserved for use as control samples and were not implanted further. The remaining dice were then given an over-all implant of 1×10^{13} 260 keV Se^{+}/cm^{2} at room temperature or at 250°C. The dice where then annealed in sets. These sets generally contained both implanted and control samples. Following annealing, the encapsulant was removed with HF, and In-Ag-Ge contacts were soldered to the corner area. Hall-resistivity measurements were made on all dice. Results from a few dice that exhibited excessive drift in their measured values are not included.

Tables 6 and 7 summarize the electrical measurements on these ion/substrate combinations. Because of degeneracy effects, the measurable donor concentration is not necessarily a quantitative representation of the actual number of active donors present.[11] Assuming a Gaussian distribution of implanted ions, the dose/energy parameters used would result in only 40% of the impanted ions being detected as donors by Hall-effect measurements even if all of the ions were made electrically active. If implant channeling and/or diffusion broaden the implanted profile, a greater activation could be measured, but only where sufficient ion movement has occured to reduce the peak concentration substantially from the theoretical starting value of $10^{18}/cm^{3}$.

Observed donor activation greater than 100% has been attributed elsewhere to the encapsulant deposition parameters.[3] Table 6 shows that greater than 100% donor activation follows higher temperature anneals, even given an allowance for the presence of conductive skin (as evidenced by data from the control samples). At 800°C, the various batches of silicon dioxide have essentially the same effects on the two ingots examined and comparisions of the other films at this anneal temperature seem justified. Encapsulant parameters were not compared for anneal temperatures greater than 800°C. Data from samples given a proprietary capless anneal is included as reference for this ingot.

Table 6. Summary of Anneal Studies on GaAs Ingot #3106

Implant: 10^{13} 260 keV Se^{+}/cm^{2} at Room Temperature or 250°C.

Encapsulant	Number of Dice	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/Vsec$	Sheet Electron Concentration, cm^2	Anneal Temperature, °C
Implanted at room temperature:					
None[a]	2	444 ± 39	3420 ± 50	$(4.14 \pm 0.42) \times 10^{12}$	800
Control	1	6.5×10^{5}	-36.30	-2.6×10^{11}	800
Low temperature oxide I	2	537 ± 38	3230 ± 100	$(3.62 \pm 0.36) \times 10^{12}$	800
Control	0	none			800
Low temperature oxide I	1	222.7	3073	9.12×10^{12}	850
Control	0	none			850
Low temperature oxide I	1	106.4	3404	1.72×10^{13}	900
Control	1	503.1	2678	4.63×10^{12}	900
Implanted at 250°C:					
None[a]	2	231 ± 29	3780 ± 420	$(7.22 \pm 0.12) \times 10^{12}$	800
Control	1	5.3×10^{7}	177.5	6.7×10^{8}	800
Low temperature oxide I	2	282 ± 18	3770 ± 40	$(5.89 \pm 0.32) \times 10^{12}$	800
Control	0	none			800
Low temperature oxide I	1	171.3	3225	1.13×10^{13}	850
Control	0	none			850
Low temperature oxid I	1	85.64	3191	2.28×10^{13}	900
Control	1	182.4	3268	1.05×10^{13}	900

[a]Capless anneal.

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TABLE 7. SUMMARY OF ENCAPSULANT STUDIES ON GaAs INGOT #2988

Implant: 1×10^{13} 260 keV Se^{+}/cm^{2} at room temperature. 800°C, 30-min anneal.

Encapsulant	Number of Samples	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2/V\text{-}sec$	Sheet Electron Concentration, cm^{-2}
Low temperature oxide I	4	454 ± 32	3360 ± 370	$(4.10 \pm 0.18) \times 10^{12}$
Control	2	1520 ± 175	4036 ± 93	$(1.03 \pm 0.14) \times 10^{12}$
Low temperature oxide II	4	402 ± 40	3206 ± 353	$(4.88 \pm 0.17) \times 10^{12}$
Control	2	982 ± 162	4135 ± 211	$(1.57 \pm 0.34) \times 10^{12}$
High temperature oxide	4	480 ± 260	3173 ± 1159	$(4.77 \pm 0.47) \times 10^{12}$
Control	1	-7×10^{5}	5280	1.5×10^{11}
High temperature oxide blended to nitride and back to oxide	4	488 ± 18	3593 ± 63	$(3.56 \pm 0.12) \times 10^{12}$
Control	2	$(1.8 \pm 0.85) \times 10^{8}$	198.4 ± 38	$(1.87 \pm 0.53) \times 10^{8}$
Thin high temperature oxide overcoated with thick nitride	2	$(1.9 \pm 1.1) \times 10^{8}$	106.2 ± 58	$(5.0 \pm 4.9) \times 10^{8}$
Control	2	$(1.69 \pm 0.5) \times 10^{8}$	164.4 ± 53	$(2.59 \pm 1.5) \times 10^{8}$
Thin high temperature oxide overcoated with thin nitride and oxide	4	478 ± 37	3107 ± 329	$(4.24 \pm 0.31) \times 10^{12}$
Control	1	8.05×10^{8}	49.34	1.57×10^{8}
Oxynitride (25% O) overcoated with high-temperature oxide	4	$(3.1 \pm 3.7) \times 10^{4}$	1526 ± 131	$(3.2 \pm 1.2) \times 10^{12}$
Control	1	7.20×10^{7}	-1.073	-8.08×10^{10}
Oxynitride (15% O) overcoated with high-temperature oxide	2	$(1.5 \pm 1.8) \times 10^{4}$	580.0 ± 734	$(3.46 \pm 0.22) \times 10^{12}$
Control	1	2.02×10^{7}	-10.52	-293×10^{10}
Thin nitride overcoated with high-temperature oxide	3	1030 ± 260	2370 ± 670	$(2.7 \pm 0.1) \times 10^{12}$
Control	2	$(5.66 \pm 1.2) \times 10^{8}$	681.6 ± 76.3	$(1.65 \pm 0.18) \times 10^{7}$
Thin nitride only	4	$(1.4 \pm 0.9) \times 10^{4}$	144 ± 193	$(4.6 \pm 3.0) \times 10^{12}$ (much drift)
Control	1	6.06×10^{7}	14.25	7.12×10^{9}
Thick nitride only	4	$(4.4 \pm 4.0) \times 10^{8}$	91 ± 77	$(1.3 \pm 1.7) \times 10^{9}$ (much drift)
Control	2	$(13.6 \pm 6.0) \times 10^{8}$	238 ± 68	$(2.4 \pm 1.6) \times 10^{7}$

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Tables 6 and 7 show that dice encapsulated with an oxide interface and that lack a thick nitride film will exhibit similar characteristics regardless of the method used to deposit that film and the ingot examined. The dice coated with oxynitrides had lower carrier mobility; the stoichiometric 600°C Si_3N_4 used further depressed the measured mobility and produced visible strain in the dice surfaces. Capless annealing produced data in good agreement with that obtained with oxide encapsulants. The measurements of these dice show 40% apparent activation, which is in good agreement with the Gibbons and Tremain argument[4] and indicates that little spreading has occurred. Dice with the same annealing conditions which had been implanted at 250°C exhibited 70% apparent activation (Table 6) and a drop in peak carrier concentration to about $10^{17}/cm^3$ (assuming 100% actual activation).

The variations in Ga vacancy concentration indicated by the significant differences in the amount of Ga detected in the encapsulant film following 800°C annealing would be expected to affect the electrical measurements, but no effects were found. No correlation was found between the electrical measurements and the permeability of the encapsulant film to Ga. The mechanical stress inherent in the Si_3N_4 used has outweighed any advantages from its lower permeability to Ga.

These results appear to contradict those of the MIT Lincoln Labs group,[1] who have had good success with pyrolytic silicon nitride. But one of their investigators, however, recently disclosed that the deposition parameters used at Lincoln Labs are significantly different from those used at HRL. In particular, the best results at Lincoln Labs resulted when gas mixtures were used that have ammonia concentrations far above those we have been using. Silicon nitride deposited under these conditions may have lower stress or higher plasticity than the films we have been using. We will evaluate films of this type early in the next contract period.

SECTION 3

SULFUR TRANSFER TEST S 1

In cooperation with Code 5212, NRL, we tested the transferability of ion implantation of sulfur into GaAs using plasma-deposited Si_3N_4 as an annealing cap. This section summarizes the results of this test. A more complete discussion is provided in Ref. 2. As in the beryllium implantation transfer test described in a previous progress report,[12] complete sets of test dice were to be processed independently at both HRL and NRL. At the time of these experiments, however, neither facility was equipped to deposit reliable Si_3N_4 films. At that time, Dr. Adir Jacob of LFE Corporation, Waltham, Massachusetts, volunteered to prepare plasma-deposited Si_3N_4 films on samples from both laboratories. We therefore decided to compromise the total separation of the samples from the two laboratories by using encapsulant films from a single source. We wish to thank Dr. Jacob for supplying the encapsulant films and Mr. William Uchytil of Metro-Line Western, Inc., Anaheim, California, for arranging to have the coatings prepared.

Before the samples were shipped to Dr. Jacob, all the dice were chemically cleaned at HRL. The Si_3N_4 films were deposited only on the implanted side of the test dice. To protect against degradation of the edges of the samples during annealing, all dice were coated on both sides with $\sim$2000 Å of pyrolytic SiO_2 at HRL before further processing. Thus, this transfer test represents a test of the transferability of the implantation and annealing procedures employed, but not of the ability of the two laboratories to prepare functionally identical Si_3N_4 coatings. The complete processing sequence used is detailed in Table 8.

To assess the consistency of the electrical evaluation techniques at our two facilities, all the samples were tested for sheet resistivity Rs ($\Omega/\square$) and Hall-effect mobility μH ($cm^2V^{-1}S^{-1}$) at both laboratories. The results of these measurements are presented in Table 9.

Statistical analysis of the electrical data was performed to determine if systematic differences existed between the samples prepared

Table 8. Implant Processing for Sulfur Transfer Test S1

Step	Description
1	Ninety-eight dice from Crystal Specialties ingot No. 3106, numbered between 1 and 100, sent to NRL. 99 dice, numbered 101 to 199, used at HRL. All dice cleaved from slices No. 13 through No. 18 and roughly 7 mm x 7 mm x 0.3 mm.
2	Dice implanted in sets of four (four dice = 1 batch). Two control dice processed with each batch and shielded from implantation by masking.
3	Pre-implant cleaning: (a) Heat in TCE to bubbling (b) Flush, thoroughly rinse, then heat to bubbling in methanol (c) Flush and thoroughly rinse in deionized water (d) Soak in HF 1 minute (e) Rinse thoroughly in deionized water (f) Blow dry with filtered boil-off nitrogen.
4	Two batches implanted with 5.6×10^{13} cm^{-2} 120 keV S^+ at room temperature.
5	Two batches implanted with 5.6×10^{13} cm^{-2} 120 keV S^+ at 250°C.
6	All samples implanted with corner contacts (a) Masks heavy aluminum foil, revealing quarter-circle in each corner of sample, radius of quarter-circle ~1/16 in. (b) Dose 5×10^{14} cm^{-2} 20 keV S^+, 5×10^{14} cm^{-2} 140 keV S^+, both at room temperature.
7	Repeat steps 3(a), 3(b), and 3(f).
8	Step 7 repeated at HRL.
9	Samples coated with roughly 2000 Å Si_3N_4 at LFE Corporation (implanted side only)
10	Samples coated on both sides with roughly 2000 Å of pyrolytic SiO_2 at HRL. Annealing performed in flowing forming gas (15% H_2, 85% N_2).
11	Anneal time 30 minutes. All glassware in hot zone of furnace is Spectrosil synthetic quartz. One batch from each of steps 4 and 5 annealed at 800°C. One batch from each of steps 4 and 5 annealed at 900°C.
12	Remove encapsulant by soaking in HF. Quench and rinse thoroughly in DIW. Blow dry with filtered dry nitrogen gas.
13	Corner contacts soldered on using In-Ag-Ge solder prepared as follows: (a) Cover most of one surface of a Ge wafer with 90:10 In:Ag solder (Indalloy No. 3) (b) Anneal in argon at 600°C until melt forms into a smooth-surfaced mass
14	Soldering performed using a fine-tipped soldering pencil. Solder alloy is simply picked off of surface of Ge wafer with soldering tip. Contacts kept as small as possible consistent with requirements of Hall measurement apparatus. Sinter contacts if necessary in forming gas for 30 sec at 300°C.

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Table 9. Composite Results from Sulfur Transfer Test "S1"

Statistics: 90% Confidence Limits on Mean Values of Measured Variables

Implant Temperature, °C	Anneal Temperature, °C	Parameter: R_S (Ω/□) or μ_H ($cm^2V^{-1}s^{-1}$)	Prepared at Lab A			Prepared at Lab B		
			Measured at					
			N*	Lab A	Lab B	N*	Lab A	Lab B
20 (Nominal)	800	R_S	3 (4)	252 ± 28 234 ± 46	253 ± 27 256 ± 17	2 3r	230 ± 80 259 ± 86	227 ± 56 257 ± 89
		μ_H	3 (4)	s3080 ± 100 3420 ± 790	3240 ± 190 3210 ± 140	2 3r	2700 ± 1000 2450 ± 870	2840 ± 760 2580 ± 800
	900	R_S	2	82.8 ± 0.1	m84.2 ± 0.6†	3	--	67.4 ± 7.8
		μ_H	2	2720 ± 190	2870 ± 470	3	--	2850 ± 390
250	800	R_S	3r (4r)	92.8 ± 4.7 94.9 ± 5.5	s93.7 ± 2.0 121 ± 64■	3 4	110 ± 20 --	113 ± 21 112 ± 12
		μ_H	3r (4r)	3730 ± 210 3700 ± 140	3870 ± 170 3400 ± 1200■	3 4	3470 ± 80 --	3650 ± 70† 3640 ± 40
	900	R_S	3r 4r	m69.1 ± 6.7 70.7 ± 5.3	m70.3 ± 5.2 --	4	90 ± 13	86.4 ± 6.0
		μ_H	3r 4r	s3340 ± 40 3380 ± 100	m3440 ± 80 --	4	3580 ± 260	3900 ± 140

*Number of samples. Superscript "r" denotes that one or more of the samples in the data set has a four-terminal resistance ratio greater than 3.0. Parentheses indicate that a measurement of one sample at one facility is questionable by inspection.

†Lab A and Lab B mean value limits on same samples do not overlap.

■Lab A and Lab B values for measurements on same samples do not pass F test for standard deviations at 90% confidence level.

mMean value limits for samples prepared at Lab A and Lab B (measured at same facility) do not overlap.

sMeasurements at same facility on samples prepared at Lab A and Lab B do not pass F test for standard deviations at 90% confidence level.

at the two facilities or between the measurements compiled at the two locations. The analysis used was described in detail in Ref. 2.

Transferability was assessed for 14 pairs of data sets: 6 for samples implanted at room temperature and 8 for samples implanted at 250°C. Of the 6 pairs of room-temperature data, one pair failed to pass our criterion for consistency of mean value, and another failed our test for consistency of dispersion. These results indicate that, for samples implanted at room temperature and annealed at eigher 800°C or 900°C, the process sequence used is transferable. On the other hand, of the 8 pairs of data for 250°C implants, there were three failures of the mean value test and two failures of the dispersion test. All three values of the mean value test occurred for samples annealed at 900°C. Thus, there is a clear failure to achieve transferability for implantation at 250°C with annealing at 900°C. On the other hand, implantation at 250°C followed by annealing at 800°C appears to be transferable.

Twenty pairs of data sets were compared for measurement consistency. Two pairs failed our test for consistency of mean values and two pairs failed the test for dispersion. This failure rate is consistent with normal statistical fluctuations of the measurements and indicates that there are no significant systematic differences between the measurements at the two facilities for tests involving few samples sets.

On the basis of these results, we conclude that the implantation and annealing process sequence that we have used is transferable for 20°C implantation followed by 800°C or 900°C annealing and for 250°C implantation followed by 800°C annealing. A failure of transferability was observed for 250°C implantation with 900°C annealing. These experiments were performed using plasma-deposited Si_3N_4 prepared by an outside source. Thus, this transfer test does not reflect the ability of our two laboratories to prepare functionally equivalent encapsulant films. Further experiments will be performed to answer this question.

Table 10 gives some indication of our ability to produce electrically active layers of high quality. The layers produced by room-temperature implantation followed by 800°C annealing were decidedly inferior to all the other layers. The remaining layers are roughly three times lower in sheet resistivity than those implanted at room temperature and

Table 10. Results for Four Implant Conditions Using Pyrolytic SiO_2 Encapsulation and 800°C, 30-min Anneals in Argon

Implanted Species	Fluence of Each Species, cm^{-2}	No. of Samples	Sheet Resistivity, Ω/□	Hall Electron Mobility, $cm^2V^{-1}s^{-1}$	Apparent Electrical Activity, %
S	10^{13}	4	420 ±68	3730 ±100	44.1 ±5.5
S + Ga	10^{13}	3	387 ±55	3740 ±76	41.4 ±5.3
S	10^{14}	2	105 ±10	3390 ±70	17.7 ±2.1
S + Ga	10^{14}	3	119 ±9	3260 ±11	16.1 ±1.2
Se	10^{13}	3	395 ±34	3290 ±450	48.8 ±5.1
Se + Ga	10^{13}	2	371 ±36	3630 ±40	46.6 ±4.9
Se	10^{14}	4	220 ±43	2777 ±83	10.4 ±1.8
Se + Ga	10^{14}	4	145 ±10	2998 ±52	14.4 ±1.1

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annealed at 800°C. In terms of carrier mobility, all the samples implanted at 250°C are definitely superior to those implanted at 20°C and annealed at 900°C. Of those implanted at 250°C, the samples annealed at 900°C have lower sheet resistivities than samples from the same facility but annealed at 800°C. No clear conclusion can be drawn concerning the relative mobilities obtained at the two anneal temperatures, however. Thus it is clear that better overall results were obtained from the samples implanted at 250°C. Considering the apparent transferability failure observed for 250°C implantation with 900°C anneals, the results obtained from this transfer test indicate that the best choice for obtaining reproducible layers of high quality from this process sequence is to implant at 250°C and anneal at 800°C.

SECTION 4

DUAL IMPLANTATION

Using dual implantation techniques, we were able to increase the relative activation efficiency of selenium donors by 40% at a dose of 10^{14} cm^{-2} (Ref. 4). Table 10 summarizes our results for four implant conditions using pyrolytic SiO_2 encapsulation and 800°C, 30-min anneals in argon. These results were obtained from implants performed at elevated temperature (200 or 250°C) and at random incidence (7° tilt) into bare GaAs substrates from Crystal Specialties ingot 2988. Dual implants, where used, were performed first and were spatially matched to the donor implant (projected ranges were matched; standard deviations of projected range agree within a few percent). The sulfur implants were performed at 100 keV and dual implants of Ga at 300 keV. Selenium implants were performed at 220 keV with dual implants at 200 keV. The values obtained for the selenium implants at 10^{14} cm^{-2} are in good agreement with the results reported by Woodcock[12] using CVD "silicon nitride" encapsulation.

These results show clearly that dual implantation with sulfur (under the conditions studied) has no significant effect other than to reduce slightly the scatter in the data (as had been observed in our previous studies). In contrast, dual implantation does significantly affect the electrical activity of selenium at the higher dose studied. Our hypothesis is that neither hot implantation nor 800°C annealing by itself is sufficient to cause strong gallium-site occupation by implanted gallium atoms, but that the lattice disruption caused by the hot selenium implant performed after the gallium implant provides sufficient steady-state disorder to encourage preferential lattice-site location by both the selenium and gallium implants. Dual implantation will be effective only if both implants exhibit strong lattice-site preference. Sulfur implantation after gallium implantation is ineffective presumably because insufficient disruption occurs during the implantation process. If this is true, additional damage generation following the gallium implant (e.g., by proton or alpha-particle bombardment) should cause enhanced electrical

activity of sulfur implants in conjunction with gallium dual implantation. Alternatively, gallium implantation following sulfur implantation may be effective. Doping profile alteration under all dual implantation conditions is to be expected.

SECTION 5

STUDIES OF THE THERMAL CONVERSION OF SEMI-INSULATING GaAs

During this contract period, we analyzed two samples of high-purity, semi-insulating LEC GaAs and one sample of Varian semi-insulating GaAs (all supplied by Code 5221, NRL) plus an unidentified wafer of semi-insulating GaAs supplied by Code 5211, NRL. All of these samples exhibited p-type conversion under our standard test procedure (2-hr anneal at $\sim 800^{\circ}C$ in a liquid-tight container submerged in a Ga melt saturated with GaAs). The container is readily permeated by the vapors from the melt, which provide an equilibrating ambient to prevent surface decomposition of the wafer). Both n- and p-type conversions have been observed under this test procedure.

The two LEC samples (from ingots II-40L and II-22) and the Varian wafer exhibited mild p-type conversion, having sheet carrier concentrations (after annealing) of 4×10^{11}, 2×10^{11}, and 4×10^{11} cm^{-2}, respectively, with corresponding Hall mobilities (for holes) of 230, 68, and 328 $cm^2V^{-1}s^{-1}$. Stripping measurements on the sample from ingot II-40L indicated that most of the electrical activity was confined to the outer 3000 Å of material, but p-type conductivity persisted even after removal of 1.2 μm of material. Stripping of the other two substrates gave inconclusive results.

The unidentified wafer from Code 5211, NRL, exhibited extremely strong p-type conversion. After our 2-hr test, this material exhibited a sheet carrier concentration of 9.4×10^{13} cm^{-2} with a Hall mobility of 259 $cm^2V^{-1}s^{-1}$. No stripping was performed on this sample.

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